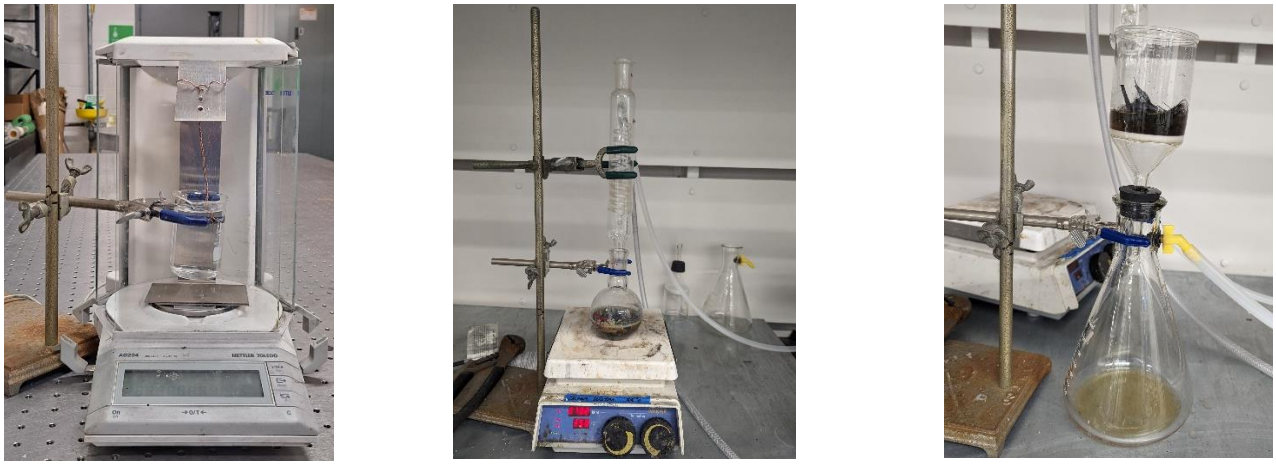
Procedure for estimating the volume and mass fraction of fiber-reinforced composites

A. Estimate of the density of the fiber-reinforced composite specimen (refer Figure 1 for measuring setup)

1. Measure the weight of the specimen in the air (M_{air}) using a weighing balance of 0.1 mg resolution.
2. Fix a tumbler filled with demineralized water in a 3-prong clamp stand and place it near the weighing balance.
3. Immerse the specimen in water and measure the weight of the specimen in water (M_{water}).
4. Specific gravity of specimen, S: $M_{air}/(M_{air}-M_{water})$
5. Density of FRP composite, $\rho_c = \rho_{water} \times S$



(a) (b) (c)

Figure 1 (a) Density estimation (ASTM D792), (b) matrix digestion, and (c) filtration/fiber extraction (ASTM D3171)

B. Estimate the mass fraction and/or volume fraction of constituents in the fiber-reinforced composite. The preparation of the potassium hydroxide (KOH) solution, matrix digestion, and fiber filtration shall be carried out in a fume hood.

1. Prepare 10% KOH solution in ethylene glycol.
   - Using a measuring cylinder, put 100 mL ethylene glycol in a 250 mL boiling flask.
   - Place a stir bar (1/2" length) into the boiling flask.
   - Add 10 g KOH to the boiling flask and gently mix it using the magnetic stirrer (at 500 rpm) maintaining 50°C temperature on the heating plate until KOH fully dissolves.
2. Put the specimen into the boiling flask containing 10% ethylene glycol solution and connect the reflux condenser to the flask. Gently raise the temperature of the hot plate to 350 °C and stir it at 700 rpm.

   Continue heating and stirring till the matrix is completely dissolved and the fibers are separated.
3. Switch off the heater and let the solution cool down to room temperature.
4. Connect the Buchner funnel (with sintered glass filter) to a 1000 mL filtering flask secured with a 3-prong clamp attached to a weighted support stand and precondition the sintered glass filter with KOH-Ethylene glycol solution.
5. Connect vacuum hose to filtering flask and set the vacuum pressure to 70 kPa.
6. Filter the contents of the boiling flask. After filtration is completed set the vacuum pressure to 20 kPa.
7. Pour 50 mL dimethylformamide (DMF) into the boiling flask and rinse it. Subsequently, use it to wash the fibers in the funnel, to make sure you transfer all the fibers (from the boiling flask) – repeat two times.
8. Wash the fibers with demineralized water – repeat three times.
9. Place the filtered fibers along with the Buchner funnel into the oven at 100 °C for 90 minutes. Subsequently, let it cool down to room temperature in a desiccator.
10. Weigh the fibers (M_f) to the nearest 0.1 mg.

11. Calculations
    - Mass fraction of the fibers, $W_r = (M_f / M_i) \times 100$
    - Mass fraction of the matrix, $W_m = (100 - W_r)$
    - Volume fraction of the fibers, $V_r = (\rho_c / \rho_r) \times W_r$
    - Volume fraction of the matrix, $V_m = (\rho_c / \rho_m) \times W_m$
    - Void fraction of the FRP composite, $V_v = 100 – (V_r + V_m)$

Note:

1. Refer Figure 1 & 2 for the density estimation and matrix digestion process, respectively.
2. The chemical solution and filtrate from the process shall only be disposed into hazardous waste bottles and shall not be discharged directly into the drain.

Reference standards:

- ASTM D3171 – Standard test methods for constituent content of composite materials
- ASTM D792 – Standard test methods for density and specific gravity (relative density) of plastics by displacement